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(पहला पुनरीक्षण)

Guide for Treatment and Disposal of Effluents of Fertilizer Industry

(First Revision)

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FOREWORD

This Indian Standard (First Revision) was adopted by the Bureau of Indian Standards after the draft finalized by the Environment Protection Sectional Committee, had been approved by the Chemical Division Council.

The fertilizer industry made a humble beginning in 1906, when the first manufacturing unit of single super phosphate (SSP) was set up in Ranipet near Chennai with an annual capacity of 6 000 MT. The Fertilizer and Chemicals Travancore of India Ltd. (FACT) at Kochi in Kerala and the Fertilizers Corporation of India (FCI) in Sindri in Bihar (now Jharkhand) were the first large sized fertilizers plants set up in the 40's and 50's with a view to manufacture fertilizers to increase food-grains production. Subsequently, green revolution in the late 60's gave an impetus to the growth of fertilizer industry in India. There was rapid expansion to capacity in the decades of 1970s, 80s and 90s to fulfil requirement of fertilizers for Indian Agriculture.

The fertilizer industry in India is one of the eight core sectors of Indian economy. India is the third largest producer and the second largest consumer of chemical fertilizers in the world. The Department of Fertilizers, under Ministry of Chemicals and Fertilizers, is responsible for adequate and timely availability of fertilizers at affordable prices for maximizing agricultural production in the country. It is categorised as one of the most polluting industries under 17 red categories of industries by the Ministry of Environment, Forest and Climate Change (MOEF & CC), Government of India.

Fertilizer industries consumes quite large quantities of water. With increased production of fertilizer in the country over last 40-50 years, there is increasing problem in availability of fresh water. The water consumed in the manufacturing process gets polluted and is either being recycled, reuse or discharged into the streams after suitable treatment to conform the prescribed standards by State/Central Pollution Control Board (CPCB) and Ministry of Environment Forests and Climate Change (MOEF&CC), Government of India.

This standard was first published in 1981. The fertilizer industry was under the phase of development in 1980s. Since then there has been considerable increase in the capacity of the nitrogenous and phosphatic fertilizers in the country.

During last 30 years, there have been significant improvement in water management in fertilizer industries through process modifications, recycle and reuse of treated effluent water, thus reducing specific water consumption per tonne of product and minimizing the effluent discharged.

There have been developments in the treatment of raw water, cooling water treatment, boiler feed water treatment and effluent treatments. The fertilizer industry stopped use of chromate based chemicals for water treatment since last two decades. Use of arsenic in CO_2 removal process was also stopped, eliminating this water pollutant as well. Thus, there is change in characteristic of effluent and hence, the treatment technologies.

The guidelines for use of waste water for irrigation purpose have been issued by the CPCB. The MoEF & CC has revised the standards for quantity and quality of waste water discharge from fertilizer plants. The key parameters like *p*H, nitrate nitrogen, and phosphate in effluent are being monitored through online monitoring system. References of tolerance limits for industrial effluents under IS standards in the earlier Guide have been omitted and prevalent discharge limits prescribed by MoEF&CC have been included.

The plants have also adopted zero liquid discharge technologies like reverse osmosis, ultra filtration and membrane bio reactor. The revised edition of the guide covers the technological developments in treatment of waste water from fertilizer plants.

A list of relevant references is given in Annex A.

The composition of technical committee responsible for formulation of this standard is given in Annex B.

Indian Standard

GUIDE FOR TREATMENT AND DISPOSAL OF EFFLUENTS OF FERTILIZER INDUSTRY

(First Revision)

1 SCOPE

The guidelines on treatment of effluents in fertilizer industries includes the brief manufacturing processes, methods of treatment, recycling, reuse and disposal of treated trade liquid effluents from nitrogenous and phosphatic fertilizer industries. It also includes various available data, information on the sources, characteristics, quantities and environmental impacts of effluents to the surrounding water bodies.

2 NITROGENOUS FERTILIZERS

2.1 Manufacturing Process

2.1.1 The straight nitrogenous fertilizers include urea, ammonium sulphate, calcium ammonium nitrate and ammonium chloride. Urea accounts for 82 percent of nitrogen consumption in the country. Ammonium sulphate is mainly obtained as by-product of caprolactam and steel plants. Ammonium chloride is obtained as by-product of soda ash plant. The share of ammonium sulphate and ammonium chloride in nitrogen consumption is only 1.2 percent and rest of nitrogen is derived from complex fertilizer plants. Production of calcium ammonium nitrate is stopped since 2014-15 due to safety and security reasons. Therefore, the guide will cover details of effluent management in urea plants under category of nitrogenous fertilizers.

2.1.2 Nitrogen in any fertilizer is derived from ammonia. To produce urea major intermediates are ammonia and carbon di-oxide. The basic raw materials for production of ammonia are fossil fuels, such as natural gas or naphtha or fuel oil. Carbon di-oxide is generated during reforming of natural gas or naphtha or partial oxidation/gasification of fuel oil during the production of ammonia. In India, all pants based on naphtha and fuel oil have been converted into natural gas-based plants.

2.1.3 The process of production of ammonia and carbon di-oxide is summarized as follows:

a) *Ammonia Plant* — Ammonia is produced from a mixture of hydrogen and nitrogen in the ratio of 3:1. The source of H_2 is hydrocarbons in the form of natural gas or desulphurized naphtha or Fuel Oil whereas the source of N_2 is the atmospheric air.

2.1.3.1 The various processes which are necessary for producing ammonia are as follows:

a) *Hydrodesulphurization* — All the first hydrocarbon feed is desulphurized in the desulphurization unit. This unit consists of three vessels, one containing the hydrogenation catalyst and two containing H_2S absorption mass. Organic sulphur compounds get converted into sulphur free hydrocarbons in the presence of hydrogen over the hydrogenation catalyst at a temperature of 380-400 °C, leaving hydrogen sulphide which is finally absorbed by zinc oxide in the absorption vessels.

H_2S Absorption – After hydrogenation, the process gas passes through ZnO absorbers. Zinc oxide catalyst (put in the absorption vessels) having normal operating temperature as 350-400 °C, reacts with hydrogen sulphide and carbonyl sulphide to form zinc sulphide:

$$ZnO + H_2S \longleftrightarrow ZnS + H_2$$

$$ZnO + COS \longleftrightarrow ZnS + CO_2$$

b) *Reforming* — In the reforming section, the gas containing necessary gas mixtures for preparation of ammonia synthesis gas is produced by catalytic reforming of a mixture of hydrocarbon with steam and air. Reactions involved are:

$$CnHm + n\,H_2O \longleftrightarrow n\,CO + (n + m/2)\,H_2 - \text{heat}$$

$$CH_4 + H_2O \longleftrightarrow CO + 3H_2 - \text{heat}$$

$$CO + H_2O \longleftrightarrow CO_2 + H_2 + \text{heat}$$

The conventional steam reforming takes place in two steps. An additional pre-reforming step is used in some plants depending on the feedstock.

c) *Primary Reforming* — The primary reformer is a radiant furnace where raw natural gas and recycle H_2 from ammonia synthesis loop is mixed with reforming steam to achieve steam to carbon ratio as 3.0-3.3 (to avoid carbon formation on the catalyst) and heated to a temperature of about 500 °C. The natural gas steam mixture is then passed through vertical primary reformer tubes filled with nickel based catalyst. The partially reformed gases then directly pass to secondary reformer.

d) *Secondary Reforming* — The partially reformed gas at 700-800 °C from primary reformer is mixed with process air and the mixture then passes over a bed of nickel-based catalyst in the secondary reformer. Process air feed required for fixing hydrogen/nitrogen ratio of 3 to 1 in secondary reformer at 33 kg/cm^2 of pressure is supplied by centrifugal process air compressor.

The necessary heat input to the primary and secondary reformer is supplied in different ways. In the primary reformer, heat is supplied as indirect heat by firing whereas in secondary reformer, heat is supplied as direct heat by mixing air into the gas. The burning gas provides heat for the rest of the reforming.

e) *Gas Purification* — The purpose of gas purification is to prepare a synthesis gas containing hydrogen and nitrogen in the ratio 3 to 1 and besides this only inert gases, such as argon in as low a concentration as possible.

f) *Shift Conversion* — Carbon monoxide is converted to CO_2 and H_2 with steam in order to increase the H_2 yield, in the two shift converters namely, high temperature (HT)/medium temperature (MT) CO conversion and low temperature (LT) CO conversion. Following reaction takes place in reactors:

$$CO + H_2O \longleftrightarrow CO_2 + H_2 + \text{heat}$$

HT/MT shift and LT shift converter contains shift catalyst consisting of specially prepared copper, zinc and aluminium oxides (having much higher activity) in a single bed. Because of very high activity of shift catalyst, it is possible to operate LTS in the low temperature range of 180-250 °C and low steam to dry gas ratio.

g) *Carbon dioxide (CO_2) Removal* — The gaseous mixture coming out of the shift converters contains about 0.25 mole to 0.5 mole percent CO. The reacted part of CO increases the H_2 yield with simultaneous formation of CO_2. After cooling of the gas and condensation of most of the water content, CO_2 is removed in the CO_2 removal unit, leaving less than 0.1 percent mole in the process gas.

Carbon dioxide removal process can be based on chemical process or physical process depending on the process licensor. In chemical absorption, carbon dioxide is removed by absorption in CO_2 absorber which a column is containing stainless steel packing material distributed in number of beds. In case of potassium carbonate based CO_2 removal system, the gas flows upwards in the absorber against a descending steam of hot aqueous potassium carbonate solution (which contains 25 percent potash by weight partly converted into bicarbonate, DEA and glycine as an activator and vanadium penta oxide as corrosion inhibitor). The solution is kept hot so as to:

1) increase the rate of absorption;
2) keep the bicarbonate in solution; and
3) to avoid heating of the solution before regeneration.

A number of plants also use amine based system (MDEA or a MDEA) for CO_2 removal. There are physical absorption methods available which are more suitable for by partial oxidation or gasification plants.

h) *Methanation* — After removal of CO_2, remaining traces of CO and CO_2 are removed in a methanator as even the small amounts of CO and CO_2 are severely poisonous to ammonia synthesis catalyst. The reactions involved in the reverse of reforming are:

$$CO + 3H_2 \longleftrightarrow CH_4 + H_2O + \text{heat}$$

$$CO_2 + 4H_2 \longleftrightarrow CH_4 + 2H_2O + \text{heat}$$

Thus, resulting in formation of methane at the expense of hydrogen. The methanator contains nickel catalyst on a ceramic base having the same characteristics as that of reforming one. Methane formation is favoured by low temperature, high pressure, and low steam content of the process to the methanator resulting in a significant temperature rise, needs lower inlet temperature and also requires the catalyst to be very active at such lower temperature.

j) *Ammonia Synthesis* — In the synthesis section, the purified gas that is, H_2 and N_2 in ratio 3 : 1 is converted into ammonia by a catalytic exothermic reaction after compression to a pressure of about 135-200 kg/cm^2 g at an optimum temperature. A considerable amount of heat is liberated (about 92.4 kJ/mole of produced ammonia) by the synthesis reaction.

$$3H_2 + N_2 \longleftrightarrow 2\,NH_3 + \text{heat}$$

Nearly all this heat is utilised for production of HP steam and preheating of HP boiler feed water.

In synthesis loop approx. 29~35 percent of H_2 and N_2 are converted to ammonia (per pass) depending upon the loop configuration, so it is necessary to recycle the unconverted synthesis gas. It is accomplished by a synthesis loop comprising equipment for HP steam production, boiler feed water preheating, cooling of synthesis effluent gas and condensation and separation of ammonia.

In the synthesis loop make up gas is introduced between the 2nd cold heat exchanger and 3rd ammonia chiller. At this point, a considerable part of ammonia produced in the converters has been condensed. The mixture of synthesis gas and liquid ammonia goes to ammonia separator where liquid ammonia is separated.

k) *Refrigeration and Ammonia Recovery* — The purpose of the refrigeration circuit is to condense the ammonia produced in the two converters in addition to cool the make-up gas and inert gas. The circuit consists of a compressor unit, a condenser, an accumulator and a number of chillers operating at three different temperatures. The ammonia recovery section removes and recovers the major part of the ammonia contained in the purge gas from the synthesis loop and the off gas from the refrigeration section. Purge gas from the synthesis loop is washed in 1st absorber leaving only 0.1 mole percent ammonia whereas the combined stream of let-down gas and inert vent gas from the refrigeration section is made to wash in the 2nd absorber. Liquid ammonia thus formed is either directly sent to Urea plant or to the Ammonia storage tanks.

2.1.4 *Urea Manufacturing Process*

Urea, the ultimate desired product is produced by synthesis from liquid ammonia and gaseous carbon dioxide at a pressure of around 160 kg/cm^{2}g and a temperature of 188-190 °C. The process are as follows.

2.1.4.1 *Urea synthesis*

In the high-pressure urea reactor, ammonia and carbon dioxide react to form ammonium carbamate, a portion of which dehydrates to form urea and water. The reactions are as follows:

$$\underset{\text{ammonia}}{2NH_3} + \underset{\substack{\text{carbon}\\\text{dioxide}}}{CO_2} \longleftrightarrow \underset{\text{ammonium carbamate}}{NH_2COONH_4}$$

$$\underset{\text{ammonium carbamate}}{NH_2COONH_4} \longrightarrow \underset{\text{urea}}{NH_2CONH_2} + \underset{\text{water}}{H_2O}$$

The fraction of ammonium carbamate that dehydrates is determined by the ratio of various reactants, the operating temperature and the residence time in the reactor. The mole ratio of ammonia to carbon dioxide is around 3.3 to 3.6 : 1.

The carbon dioxide coming directly from ammonia plant is compressed in a compressor. The liquid ammonia coming from the battery limit maintains the level in the ammonia receiver from where it is drawn and pumped to about 22 to 24 kg/cm^{2}g by booster pumps. A part of this ammonia goes to a medium pressure absorber and remaining ammonia is pumped to about 250 kg/cm^{2}g pressure, preheated hot condensate and is used as driving fluid in the carbamate ejector. The liquid mixture of ammonia and carbamate reacts with carbon dioxide inside the reactor.

A small controlled quantity of air is added along with CO_2 at the compressor in order to passivate the stainless-steel surfaces to protect them from corrosion due to reagents and reaction products.

The condensing of the gaseous stream permits production of steam at 4.5 kg/cm^{2}g in carbamate condenser. The un-condensable gases from the top of the carbamate separator/scrubber, consisting mainly of inert gas with a small quantity of NH_3 and CO_2 are passed under pressure control to the medium pressure section.

In the urea reactor, the reaction is complete by 60-65 percent and due to recycling of unconverted reactants, urea concentration in the product coming out of reactor is 30-35 percent.

The unconverted reactants are separated out from urea solution by reducing pressure and supplying external heat by steam, usually accomplished in the three stages namely, synthesis pressure, medium pressure 16 to 18 kg/cm^{2}g and low pressure 2.5 to 3.5 kg/cm^{2}g. The vapours so generated are condensed to obtain carbamate solution and the process is highly exothermic. Utilization of heat of condensation of carbamate vapours is the genesis of improving thermal efficiency of urea process.

The reactant product leaving the reactor are decomposed in stripper which operates at the same pressure as reactor. The urea, ammonia, carbon dioxide and ammonium carbamate mixture pass through the stripper tubes in the form of liquid film from the top, which excess ammonia or carbon dioxide (as per process technology) carries out the stripping. The overhead vapours from the stripper are recovered as ammonium carbamate in the condenser which also operates at reactor pressure. The heat of condensation in the carbamate condenser is recovered in the form of LP (4 to 5.5 kg/cm^{2}g) steam.

2.1.4.2 *Urea purification and low-pressure recoveries*

Urea purification takes place in three stages at decreasing pressure as follows:

1) *1st Purification and Recovery Stage at Medium Pressure*

   The solution with a low residual CO_2 content, leaving the bottom of the stripper is expanded at the Medium Pressure (MP) and enters the falling film type decomposer. MP decomposer consists of a flash vessel to recover flashed gases and a decomposition section where the residual carbamate is decomposed by indirect heating The NH_3 and CO_2 rich vapours from top flash separator are sent to MP absorber where they are absorbed with aqueous carbamate solution coming from LP recovery section. Bottom solution from the MP absorber is recycled back to synthesis section.

   MP pre-decomposer has been introduced as a revamp measures in some plants. The MP pre decomposer provides an initial decomposition of carbamate flowing from the Stripper bottom before entering the existing M.P. decomposer.

2) *2nd Purification and Recovery Stage at Low Pressure*

   The urea solution leaving the MP decomposer is expanded at low pressure and enters the falling film type LP decomposer. The LP decomposer consists of a top separator to flash off gases and the solution enters the tubes of the decomposer section where the last traces of residual carbamate is decomposed by means of heat supplied by steam. The gases leaving the top separator are first sent to the LP absorber/ condenser where they are absorbed in one aqueous carbamate solution coming from process condensate treatment section. Inert gases, washed in the low pressure inerts washing tower, are collected to vent stack practically free from ammonia.

3) *3rd Purification and Recovery Stage at 0.35 kg/cm^{2}a Pressure*

   The solution leaving the bottom of low-pressure decomposer is expanded at 0.35 kg/cm^{2}a and enters the falling film type vacuum pre-concentrator. It consists of a top separator, where the released flash gases are removed before the solution enters the tube bundle and decomposition section where the last residual carbamate is decomposed and the required heat is supplied by the condensation of the gases coming from the MP decomposer. A number of plants have added pre-concentrator as a part of revamp.

2.1.4.3 *Urea concentration*

To concentrate the urea solution, two stage vacuum system has been provided. The solution leaving the pre concentrator/low pressure decomposer bottom is sent to the first vacuum concentrator. The mixed phase coming out of it enters the first vacuum separator. Here, the remaining ammonia, CO_2 and mainly water are separated from top of the separator and sent to vacuum surface condenser. 95 percent urea solution from first vacuum separator enters the second vacuum concentrator. The mixed phase coming out of second vacuum concentrator enters the 2nd vacuum separator. Here 99.7 percent urea solution is sent to Prilling bucket.

2.1.4.4 *Urea prilling*

The melted urea leaving the second vacuum separator is sent to the prilling bucket by means of urea melt pumps. The urea coming out of the bucket in the form of drops falls along the prilling tower and encounters a cold air flow which causes its solidification. The heated air with a few ppm of urea dust comes out of the prilling tower top to atmosphere. The solid prills falling to the bottom of the prilling tower are sent to the belt conveyor for packaging in the packaging plant.

2.2 Effluent Generation and Treatment Guidelines in Nitrogenous (Urea) Fertilizer Plants

2.2.1 *Source of Effluent Generation*

2.2.1.1 *Ammonia-urea plants*

The major potential liquid pollutants generated from the ammonia and urea plants are process condensates, regeneration water, water treatment plant, cooling tower blow down and boiler blow down (steam generation plant). Various categories of effluents generated in the nitrogenous fertilizer complex and broadly classified as below:

a) Non-treated effluents — Effluents generated but not treated at source:
   1) Rain water/storm water generated from non-polluting areas;
   2) Boiler blows down water (steam generation plant/captive power plant); and
   3) Cooling tower blow down from ammonia and urea cooling towers.
b) Effluent generated and treated in the individual plant battery limits:
   1) Raw water pre-treatment plant/DM plant regeneration effluents;
   2) Ammonia plant process condensate;
   3) Urea plant process condensate;
   4) Turbine steam condensate; and
   5) Oil leakages/spillages from ammonia, urea and other plants.
c) Effluent treated in effluent treatment plant:
   1) Ammonia and urea process condensates in case of process upsets;
   2) Floor washings/process drains from ammonia and urea plants;
   3) Oil leakages/spillages from ammonia and urea plants; and
   4) Sanitary waste from industry and associated township.

2.2.2 *Treatment Schemes*

2.2.2.1 *Raw water pre-treatment*

Where source of water is surface water, a pre-treatment scheme is provided to remove impurities like suspended solids, microbiological organisms and also to reduce the total dissolve solids, permanent hardness etc.

A clarifier of suitable capacity is installed near the source of surface water or at the manufacturing site to remove the undesirable impurities.

Before the water enters the clarifier, coagulation and flocculation reagents, such as polyelectrolytes and Ferric/non-ferric alum can be added. These reagents cause finely suspended particles to clump together and form larger and denser particles, called flocs that settle more quickly and stably. This allows the separation of the solids in the clarifier to occur more efficiently and easily; aiding in the conservation of energy. Isolating the particle components first using these processes may reduce the volume of downstream water treatment processes like filtration. The clear water with reduced turbidity is collected as overflow channel into a clear water storage tank and the settled sludge is separated from the bottom periodically. The clear water further sent to water pre-treatment plant to reduce the permanent hardness through lime treatment if required or to the demineralization plant (DM) for further treatment. The bottom sludge is periodically removed from clari-flocculator and dried sludge is removed through vacuum drying while separated water is recycled in the process. Table 1 provides typical characteristic of raw water before and after clarifier.

Table 1 Typical Characteristic of Raw Water Before and After Clarifier

(*Clause* 2.2.2.1)

Sl No. (1)	Sample Source (2)	pH (3)	Turbidity (4)	Free Chlorine (5)
i)	Clarifier inlet	7.0 – 8.5	10-1000 NTU	NIL
ii)	Clarifier outlet	6.5 – 8.5	5-10 NTU	0.10 – 0.30 ppm

2.2.2.2 *Demineralization plant*

Demineralization of raw water is needed to produce polish water which is used as boiler feed water. The demineralization plant (DM Plant) is designed on the basis of cationic and anionic load of raw water and consists of numbers of each of weak acid cation (WAC) and strong acid cation (SAC) exchangers in series, weak base anion (WBA) and strong base anion (SBA) exchangers in series and mixed bed (MB) units. After a cycle of operation these resin beds are regenerated to remove all salts exchanged by resin through ion exchange process. The weak acidic and alkaline effluents thus coming out are collected in separate acidic and alkaline pits respectively. The quality and quantity of effluents depends upon the total dissolved solids in the raw water which controls the ion exchanger's ionic loads and through puts. These acidic and alkaline effluents are mixed proportionately and neutralized in a neutralization pit. After proper neutralization that is, *p*H is obtained between 6.5-8.5 and TDS as high as 1 500 – 3 000 ppm depending upon the TDS in raw water and the design of DM plant.

Filter beds (sand filters and activated carbon filters) are provided in upstream of DM plant to treat raw water and other streams of treated effluents being recycled from process plants to remove traces of suspended solids and organic matters (for example, methanol in ammonia process condensates). These filter beds are back washed after a cycle of operation and the effluent during the back wash is collected in the neutralization pit.

The effluent from neutralization pit is pumped to effluent pond (guard pond) *via* settling chamber or alternately stored in the effluent storage pond. An on-line *p*H meter is provided at the discharge line to ensure pH in the range of 6.5 to 8.5. Table 2 gives the typical waste water characteristics in acidic, alkaline and neutralization pits.

Table 2 Typical Waste Water Characteristic in Acidic, Alkaline and Neutralization Pits

(*Clause* 2.2.2.2)

Sl No. (1)	Sample source (2)	*p*H (3)	TDS (4)
i)	Acidic pit	0 – 7.0	2210 mg/L
ii)	Alkaline pit	7.0 – 14	1316 mg/L
iii)	Neutralization pit	6.5 – 8.5	1500 – 3000 mg/L

2.2.2.3 *Ammonia plant process condensate (APC)*

In the steam reformation process, steam excess than stoichiometric requirement is supplied in the primary/secondary reformer. After reforming and shift reactions in CO shift converter section, the excess steam in the process gas along with some of the pollutants like methanol, ammonia and water formed during methanation is separated by cooling the process gas. The condensate coming out after cooling the process gas contains impurities such as ammonia and methanol. In addition to this, in the carbon dioxide recovery section, though the absorption towers are operated in closed circuit, some absorbent chemical find their way out in the cooler concentrate and this condensate contains dissolved CO_2.

The polluted process condensates contain about 100-500 ppm of ammonia, 400-600 ppm of dissolved CO_2 and 500-1000 ppm of Methanol (Table 3).

All combined process condensates are fed to CO_2 stripper and then to steam stripper where ammonia, methanol and CO_2 are recovered as top products and recycled back to the system (Fig. 1). The stripped condensate is cooled to about 50 °C prior to delivery to the DM unit to make it boiler feed water quality. The quantity of stripped bottom product depends upon the plant capacity and the operation load.

The treated condensates contain less than 10 ppm of ammonia, 10 ppm of CO_2 and 20 ppm of methanol. The treated condensates after minor treatment through activated carbon beds to remove trace impurities is further polished through ion exchange to use it as boiler feed water.

Table 3 Typical Characteristic of APC at Inlet and Outlet of Steam Stripper

(*Clause* 2.2.2.3)

Sl No.	Sample Source	Total Ammonia as NH_3 (ppm)	Methanol As CH_3OH (ppm)	Carbon Di-oxide as CO_2 (dissolved) (ppm)
(1)	(2)	(3)	(4)	(5)
i)	Steam stripper Inlet	100-500	500-1000	400-600
ii)	Steam stripper outlet	< 10.0	< 20.0	< 10.0

FIG. 1 AMMONIA PROCESS CONDENSATE STRIPPER

2.2.2.4 *Urea process condensate (UPC)*

The process condensates generated from different sections of the urea plant contains various impurities like NH_3-4.5 to 5.0 percent, CO_2-1.90 to 2.0 percent and urea-0.80 to 1.0 percent. Quantity of such polluted process condensates depends upon the plant load.

2.2.2.4.1 *Treatment at source (within urea plant)*

Waste water containing ammonia, carbon dioxide and urea coming out from 1st and 2nd vacuum system is collected in tanks, solution is preheated and fed to distillation tower upper part (Fig. 2). After stripping in upper part of the distillation tower, the solution is preheated and fed to urea hydrolyser, where urea is decomposed at high pressure and temperature into ammonia and CO_2 and stripped off from the solution. Solution, practically free from urea is again fed to bottom part of distillation tower to remove ammonia and CO_2 present in the solution. In the distillation column where ammonia and carbon dioxide are stripped by means of saturated steam. The vapours (ammonia, carbon dioxide and water) coming from the top of the tower is condensed in overhead condenser and collected in a carbamate solution accumulator. A part of this solution is recycled back to the top of the distillation tower as reflux and the remaining is sent to the urea plant.

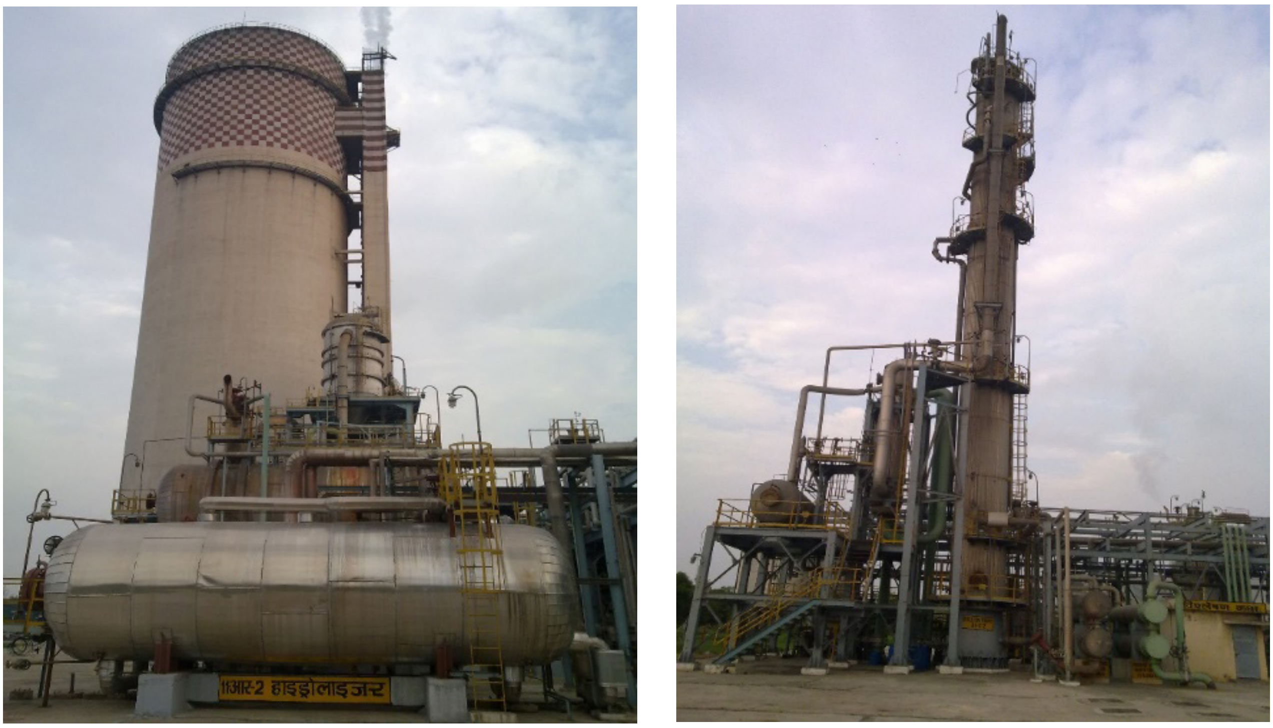

FIG. 2 HYDROLYSER AND DISTILLATION TOWER

The bottom product coming out from the distillation tower contains a maximum 5 ppm of ammonia and 5.0 ppm of urea each. This condensate is sent to DM plant for minor treatment and polishing to make it suitable for the boiler feed as per Table 4.

In hydrolyser the urea is hydrolysed into ammonia and carbon di-oxide:

$$NH_2CONH_2 + H_2O\ NH_3 + CO_2$$

Urea Steam Ammonia Carbon di-oxide

Provision of on-line analyser for Ammonia and Urea is made available to ensure the treated process condensate quality conforms to the demineralization plant requirements

Table 4 Typical Characteristic of UPC at Inlet and Outlet of Waste Water Section

(*Clause* 2.2.2.4.1)

Sl No.	Sample Source	Ammonia as NH_3	Urea as NH_2-CO-NH_2
(1)	(2)	(3)	(4)
i)	Process condensate distillation tower inlet	4.5 – 5.0 percent vol	0.80 -1.0 percent by wt
ii)	Process condensate distillation tower outlet	5.0 ppm (*Max*)	5.0 mg/L (*Max*)

2.2.2.5 *Steam and turbine condensate from ammonia and urea plants*

Steam used in the ammonia and urea plants for the purpose of maintaining temperatures get condensed after the use and normally does not get contaminated with any process materials. Similarly, the steam used for driving the turbines in the process plants are condensed after use and this also do not get contaminated with any process materials. Both these condensates are as good as DM water and recycled in process as boiler feed water after minor treatment in activated carbon filter and basket strainer to remove foreign particles in DM plant (Fig. 3).

2.2.2.6 *Effluent treatment at ETP*

Process condensates treatment during process upsets in ammonia/urea plants. In case the quality of condensate from ammonia and urea plants is off specs to be sent to DM plant for polishing due to temporary process disturbances, a provision is made to store the condensate into ammonia and urea process condensate tanks either within plant or in ETP area. Provisions are also made to transfer it back to respective plants' steam stripper/distillation tower (Fig. 4) for treatment and recycling in the process. Alternatively, when the contamination (ammonia) in the condensate is high (50 – 500 ppm) it can be stripped in steam stripper at ETP, and the stripped condensate is then pumped directly to the treated effluent pond or via available treated effluent tank. Table 5 presents typical waste water quality at inlet and outlet of steam stripper in ETP plant.

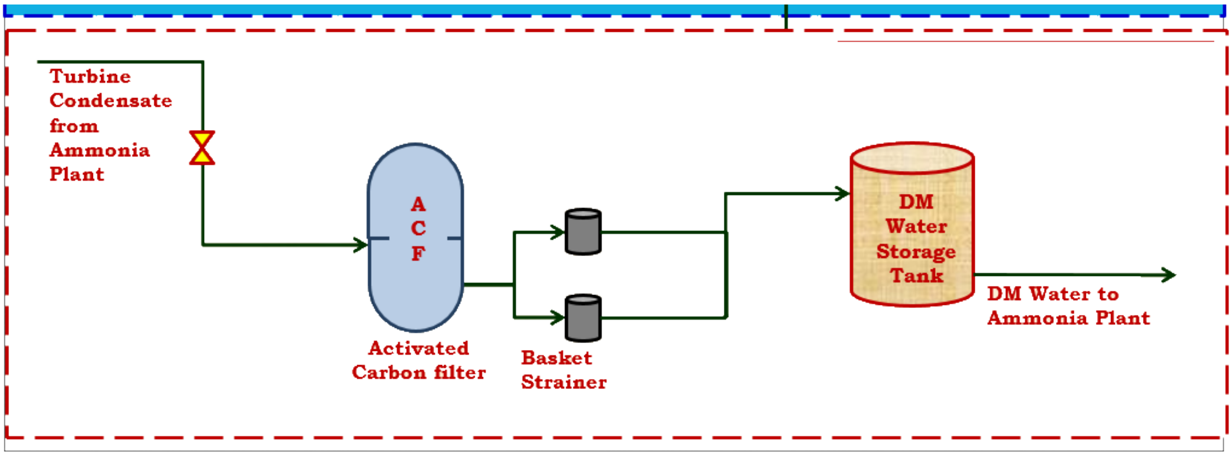

Fig. 3 Turbine Condensate Treatment Scheme from Ammonia Plant

Table 5 Typical Quality of Waste Water at Inlet and Outlet of Steam Stripper in ETP Plant

(*Clause* 2.2.2.6)

Sl No.	Sample Source	Total Ammonia as NH_3 (ppm)	*p*H	Free Ammonia As NH_3 (ppm)
(1)	(2)	(3)	(4)	(5)
i)	ETP steam stripper inlet	50-500	8.5-9.5	7.5-250
ii)	ETP steam stripper outlet	< 50	7.0-8.5	< 7.5

2.2.2.7 *Floor washings/process drains in ammonia and urea plants*

Floor washing of the plant area is carried out to remove spills and leakages from processes, however, the quantity of floor washing water is quite low. A few plants are practicing dry cleaning of shop floors in bagging plants to reduce waste water generation and load on ETP.

Floor washings of ammonia plant and urea plants are stored in the floor washing pit of the respective plant, if the quality of effluent is good (total ammonical nitrogen is less than 50 ppm and total Kjheldahl nitrogen (TKN) less than 75 ppm) then it is discharged to the guard pond directly without any treatment, if the quality of effluent is not good and concentration of ammonia is very high (50 - 500 ppm) and TKN more than 75 ppm, it is then sent to effluent treatment plant and stored in untreated tanks. Contaminated effluent from these tanks is sent to ETP steam stripper for removal of ammonia from the top and the bottom treated effluent containing < 50 ppm of total ammonical nitrogen (TAN) is discharged to the treated effluent guard pond (GP) (Table 6).

In case of accidental leakages/drains of GV solution (comprising 26-28 percent K_2CO_3, 5-6 percent $KHCO_3$, 0.5 percent glycine, 0.5 percent di-ethanol amine and 0.5 percent vanadium) a lined neutralization pit is provided with a facility of dosing of acidic $FeSO_4$ to reduce pentavalent vanadium to trivalent. Such treated effluent is sent to guard pond in a controlled manner to ensure that quality of final treated effluent (guard pond outlet) conform to the prescribed standards.

Table 6 Typical Characteristics of Waste Water Before and After Steam Stripper Outlet

(*Clause* 2.2.2.7)

Sl No.	Sample Source	Total Ammonia as NH_3 (ppm)	Free Ammonia as NH_3 (ppm)
(1)	(2)	(3)	(4)
i)	Floor washing pit ammonia/urea plant	< 100	< 15
ii)	Steam stripper outlet (ETP)	< 50.0	< 5

2.2.2.8 *Oily leakages/spillages from ammonia and urea plant*

The run-off water from compressor house, pump houses, inter-stage separators of compressors is normally polluted with oily water along with oil spillage from refrigeration systems. Waste water containing TSS as 300 ppm, ammonia 50-100 ppm and oil as 1 000 ppm is separately collected in an oily water pit provided both in ammonia and urea plants and is pumped to oil separator in ETP for removal of oil and suspended solids. The oil and emulsified suspended solids are removed through an oil skimming process, where removed oil from the top surface is stored in containers. Clear water containing less than 10 ppm oil and grease is further treated in steam stripper for removal of contaminant like ammonia, and is pumped to treated effluent pond (GP).

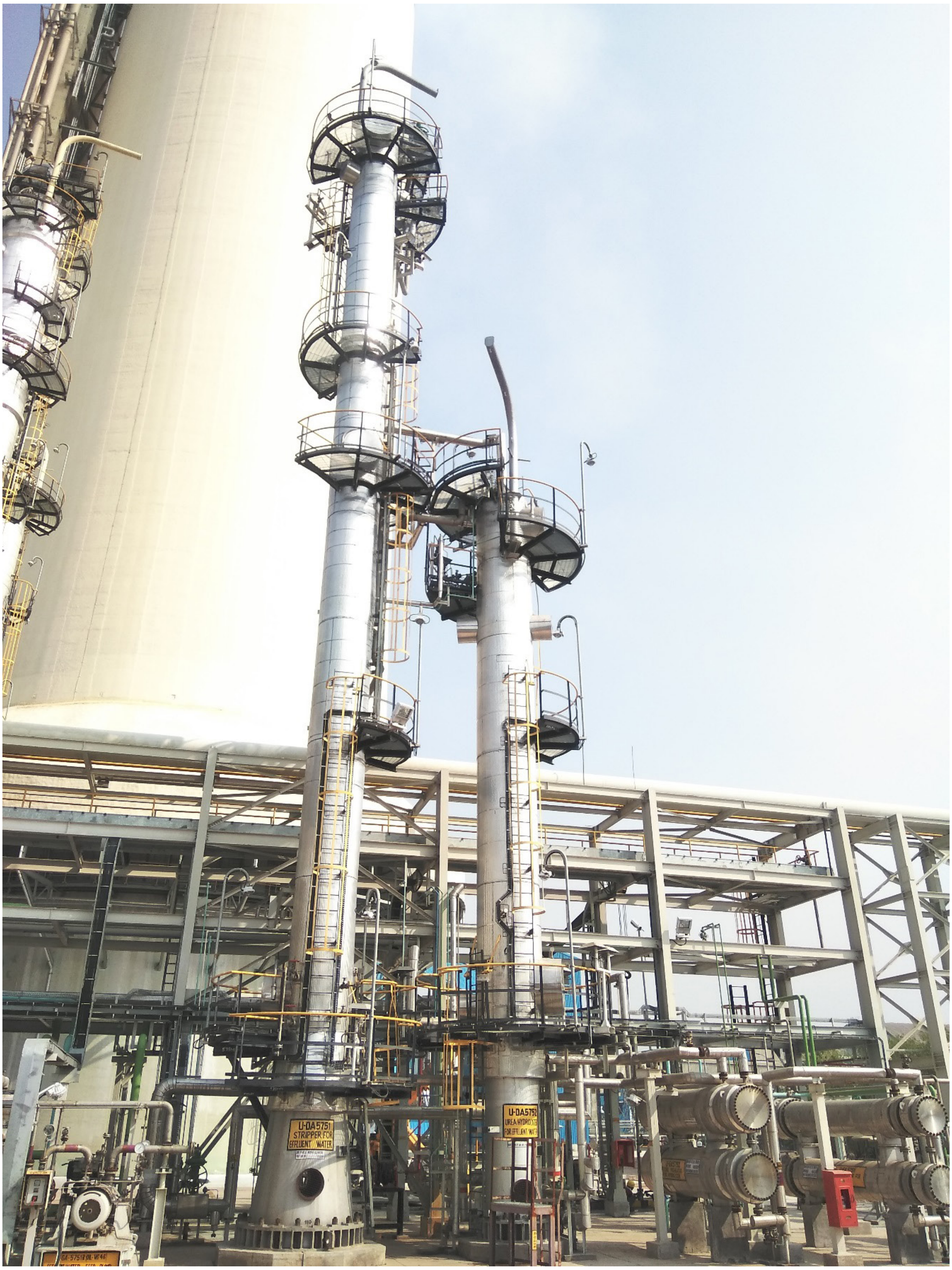

Fig. 4 View of Distillation Tower for Treating Urea Process Condensates

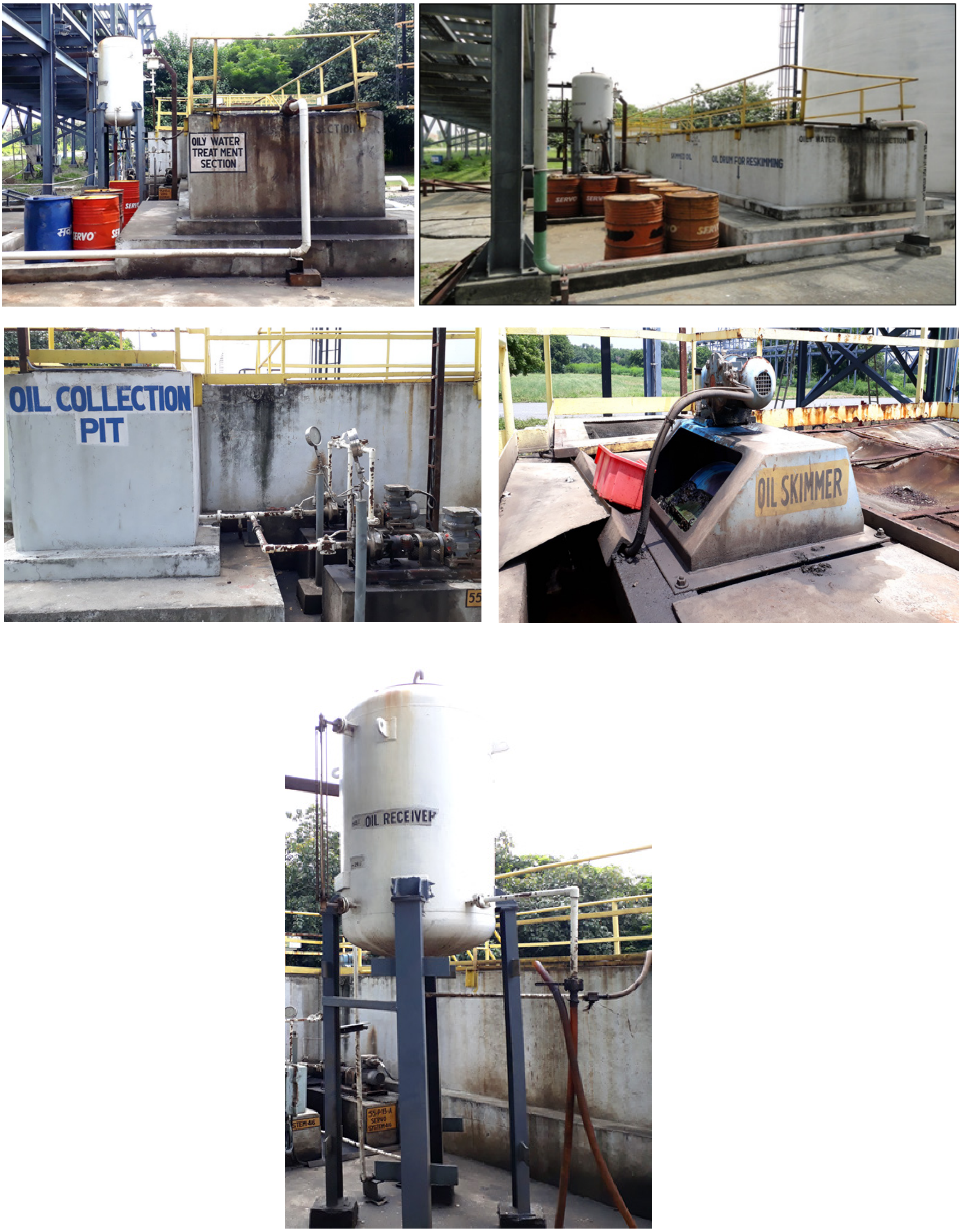

FIG. 5 OIL WATER TREATMENT PLANT

2.2.2.9 *Oil separator (ETP) treatment system*

Oily water from the oily water pits in ammonia and urea plants is pumped to oil separators where oil removal is done by an oil skimmer (Fig. 5). It is a rotary drum placed axially parallel to the surface of water in the pit. The level of the effluent in the pit is maintained by under flow weirs in such a way that the drum is always slightly immersed in the effluent. As the drum rotates, oil sticks to its surface which is coated with special features to adsorb oil and is then scraped away by a rubber scraper. The separated oil is collected in the oil pit and treated effluent is sent to collection pit from where it can either be stored in a tank if it contains total ammonia more than 50 ppm or directly sent to effluent guard pond if it contains less than 50 ppm total ammonia (Table 7). The skimmed oil is transferred to oil drums by pump. Spent oil drums from oil separation area are sent to spent lube oil storage yard (hazardous waste storage yard), from where it is sold to the authorized venders registered with applicable statutory authority.

Table 7 Typical Quality of Waste Water Before and After Oil Skimmer

(*Clause* 2.2.2.9)

Sl No. (1)	Sample Source (2)	Oil and Grease mg/L (3)	Total Suspended Solids mg/L (4)
i)	Oily water treatment inlet	10-100	10-300
ii)	Oily water treatment outlet	< 10.0	< 50.0

2.2.2.10 *Cooling water blow down and filter backwash*

Cooling water is used in the ammonia, urea and captive power plants to cool the process gas and other fluids to meet the process requirements. Cooling water requires chemical and micro-biological treatment to control corrosion in pipe lines and the process heat exchangers. These cooling towers are operated with specific number of cycles to ensure that total dissolved solids and other parameters remain within design range of parameters. To maintain parameters within specific range a continuous blow down is necessary and the waste water coming out from the blow down is discharged to the treated effluent guard pond.

The characteristic of cooling tower blow down water is given in Table 8.

Table 8 Typical Characteristic of Cooling Tower Blow-Down Water

(*Clause* 2.2.2.10)

Sl No. (1)	Parameters (2)	Value (mg/L except *p*H) (3)
i)	*p*H	6.7-7.8
ii)	Total dissolved solids (TDS)	1500-3000
iii)	Total Suspended Solids (TSS)	20-100
iv)	Zinc as Zn	2.0 (*Max.*)
v)	Phosphate as P	5-10
vi)	Nitrate nitrogen as N	5-10
vii)	Free chorine as Cl	0.5 (*Max.)*
ix)	Biological oxygen demand (BOD)	5-30
x)	Chemical oxygen demand (COD)	10-100

Value of these parameters may vary depending upon cooling tower make up water quality and number of cycles.

The blow down water is mixed with other streams of effluents coming from the plants and equalized in the guard pond for some time before finally discharged on land for irrigation or to water streams/water bodies or subjected for further treatment in ZLD plant.

Part of circulation water is also subjected to filtration through filter beds to remove impurities, such as suspended solids including organic matters. These filters are also back washed after a cycle of operation and the effluent coming out during the back wash is settled in settling pits before discharging to the guard pond.

2.2.2.11 *Boiler water blow down*

To maintain specific quality of boiler water a continuous blow down is maintained in steam generation plant and waste heat boilers. A typical characteristic is given in Table 9.

This water is sent to DM plant neutralization pit for final discharge to guard pond or may be recycled as cooling tower make up water.

Table 9 Typical Characteristic of Boiler Blow Down Water

(*Clause* 2.2.2.11)

Sl No. (1)	Parameter (2)	Value (3)
i)	*p*H	9.5 – 10.5
ii)	Silica as SiO_2	1.0 – 1.5 ppm
iii)	Phosphate as P	6.0 – 10.0 ppm
iv)	Total ammonical nitrogen (TAN) as N	< 5.0 ppm
v)	Total dissolved solids (TDS)	< 10 ppm

2.2.2.12 *Guard pond*

Guard pond is RCC structure with light density polyethylene blanket over the foundation soil rich in clay patches to ensure that pollutants do not leach underground over a period of storage and contaminate the ground water (Fig. 6). Provision of two or more guard ponds is made so that one is receiving the effluents from plants and ETP while other is used for the conditioning of the treated effluents for about one day. Treated effluent is also subjected to aeration to provide more oxygen and to control biological oxygen requirement, Eutrophication due to rich in nutrients like phosphates and nitrates etc. Continuous on-line *p*H meter and ammonia analyser is provided at the discharge of treated effluent to ensure that it conforms to the prescribed standards.

Provision of emergency pond may be considered for the storage of contaminated effluents in case of process upsets are for longer time and effluent quantity is more than the untreated effluent tanks capacity provided in the plants/ETP.

As the treated effluent from guard pond is ultimately discharged into the water streams or on land for irrigation, samples of treated effluent from guard pond is analysed for relevant parameters and as prescribed by statutory authorities like SPCB, CPCB and MOEFCC like *p*H, total ammonia, free ammonia, total Kjeldahl nitrogen, nitrate nitrogen, oil and grease, vanadium, total dissolved solids, total suspended solids, zinc, phosphate, BOD, COD etc.

A typical characteristic of final treated effluent discharge from GP is given in Table 10.

Table 10 Typical Characteristics of Treated Effluent from Guard Pond Discharge

(*Clause* 2.2.2.10)

Sl No. (1)	Parameters (2)	Value, in mg/L except *p*H (3)
i)	*p*H	6.5 – 8.5
ii)	Total ammonical nitrogen as N	50.0 *Max.*
iii)	Free Ammonical Nitrogen as N	2.0 *Max.*
iv)	Total Kjehldals nitrogen as N	75.0 *Max.*
v)	Nitrate nitrogen as N	10.0 *Max.*
vi)	Phosphate as P	5.0 *Max.*
vii)	Zinc as Zn	5.0 *Max.*
viii)	Total suspended solids	100.0 *Max.*
ix)	Chemical oxygen demand	250.0 *Max.*
x)	Biological oxygen demand	30.0 *Max.*
xi)	Cyanide as CN	0.10 *Max.*
xii)	Oil and grease	10.0 *Max.*

2.2.2.13 *Total recycle and zero effluent discharge (ZLD) plant*

Considering the water scarcity and issues related with the discharge of treated effluent on land or water streams/water bodies, provision of total recycling of effluents is made through reverse osmosis (RO) plant.

2.2.2.14 *Typical process of RO-ZLD in fertilizer plant*

The effluent from fertilizer plants is pumped to the stilling chamber with oil and grease trap. A slotted pipe skimmer is provided to remove the floated oil

Fig. 6 A Typical View of the Guard Pond

layer. Sodium hypochlorite dosing is done at the inlet of oil and grease trap. Effluent from stilling chamber outlet is entered in the flash mixing chamber; here lime/dolomite solution, soda ash and coagulant are dosed for silica and hardness reduction. The solids formed during the chemical reaction are separated in the clarification zone of downstream high rate solids contact clarifier (HRSCC). Polyelectrolyte (PE) solution with suitable concentration is dosed at the inlet chamber of HRSCC. The clarified water from the outlet of HRSCC is collected in the clarified water tank and pumped to the dual media filters and activated carbon filter followed by ultrafiltration for further treatment.

Separate backwash pumps are provided for backwash of the dual media filter (DMF). Activated carbon filter (ACF) is backwashed with feed water. Ultrafiltration (UF) permeate water is used for DMF backwash purpose. The DMF and ACF backwash waste is routed to waste recycle tank. Common air scour blower is provided to air-scour the filter media before the backwash and sludge pit.

The settled sludge from HRSCC is periodically drained to the sludge pit. Sludge from sludge pit is pumped to a centrifuge for further sludge dewatering. Centrate from centrifuge is led to flash mixing chamber. The dewatered sludge from centrifuge outlet is disposed to treatment storage and disposal facility (TSDF).

Filtered water from the ACF is fed to ultra filtration (UF) unit to reduce the fine suspended/colloidal impurities. Provision of FeCl3 dosing prior to basket strainer inlet is made. UF unit is back flushed automatically based on pre-set frequency. The UF membranes are back-flushed by UF permeate water. Permeate from the ultra-filtration system is collected in UF permeate tank. During the backwash of the UF stream, the feed water to DMF is re-circulated to clarified water tank through DMF feed pump. The UF normal backwash waste along with continuous reject is routed to the waste recycle tank. UF chemical enhance back wash (CEB) waste water is routed to UF CEB waste collection Tank. The waste from UF chemical enhance backwash (CEB) waste collection tank is pumped to flash mixing chamber after neutralization as required.

UF permeate from UF permeate tank is passed through the micron cartridge filter of 5 micron nominal rating for further fine (micron) filtration prior to RO inlet.

Provision of sodium meta bi-sulphite dosing prior to cartridge filter is made for de-chlorination. Also provision for dosing of antiscalent and Hydrochloric acid is made prior to cartridge filter. The cartridge filtered water is pumped to the RO-I block using HP pumps. A low pressure switch is provided at the suction of high pressure pump to avoid dry running. Also a high pressure switch is provided at the discharge of HP pump to avoid over pressurization. These interlocks are provided to help tripping of the system in case of occurrence of any of the above mentioned abnormalities. Permeate from RO-I is collected to common RO Permeate tank. RO-I reject is then led to RO-I reject tank and passed through RO-II skid by reject RO-II feed pump.

RO-I reject is passed through the micron cartridge filter of 5 micron nominal rating for further fine (micron) filtration.

Permeate from RO-II is collected to RO permeate tank. This permeate is used as cooling tower make up.

RO reject from RO-II is concentrated in five effect evaporator (multi) followed by pusher centrifuge. Feed is flow in forward feed manner in evaporators. All effects are forced dirculation type. Whole plant runs under vacuum.

RO reject from multi effect evaporator (MEE) feed tank is transferred by MEE feed pumps. The feed is preheated by series of pre heaters. First the feed enters the preheater–1 where the feed is heated by process condensate.

From preheater-I feed enters to next preheater-2 where the feed is heated by the partial vapors from calendria -5 and so on.

After preheater-6 the feed enters into the first effect in duct of vapor liquid separator for effect-1. When the desired level in VLS-1 is reached than RO reject is kept in recirculation by starting pump to maintain tube side velocity and to achieve desired heat transfer coefficient. The same pump is used for recirculation and to transfer RO reject to next effect. Similarly when the desired level in VLS-2 is reached the RO reject shall be transferred to next stage up to 5th stage.

Finally, RO reject is collected in Thickener tank, where pusher centrifuge is separate out salts and mother liquor. Mother liquor is recycling back to 5th stage VLS.

Dry saturated steam of 3.0 kg/cm^2 (g) is fed to the inlet of TVR as a motive fluid. Partial vapors from Effect-1 is sucked by TVR to mix up with live steam which is given to shell side of 1st effect calendria. The effluent mass which is in recirculation through calendria 1 is flashed in vapor liquid separator-1.

The vapors generated in vapor liquid separator-1 is go for next calendria-2 and so on. Finally vapor from VLS-5 is entering in surface condenser where vapor is condensed by cooling water and is mixed up with process condensate of calendria 5. This is final product of ZLD plant. A typical inlet and reject quality at RO-1 and RO-2 is given in Table 11.

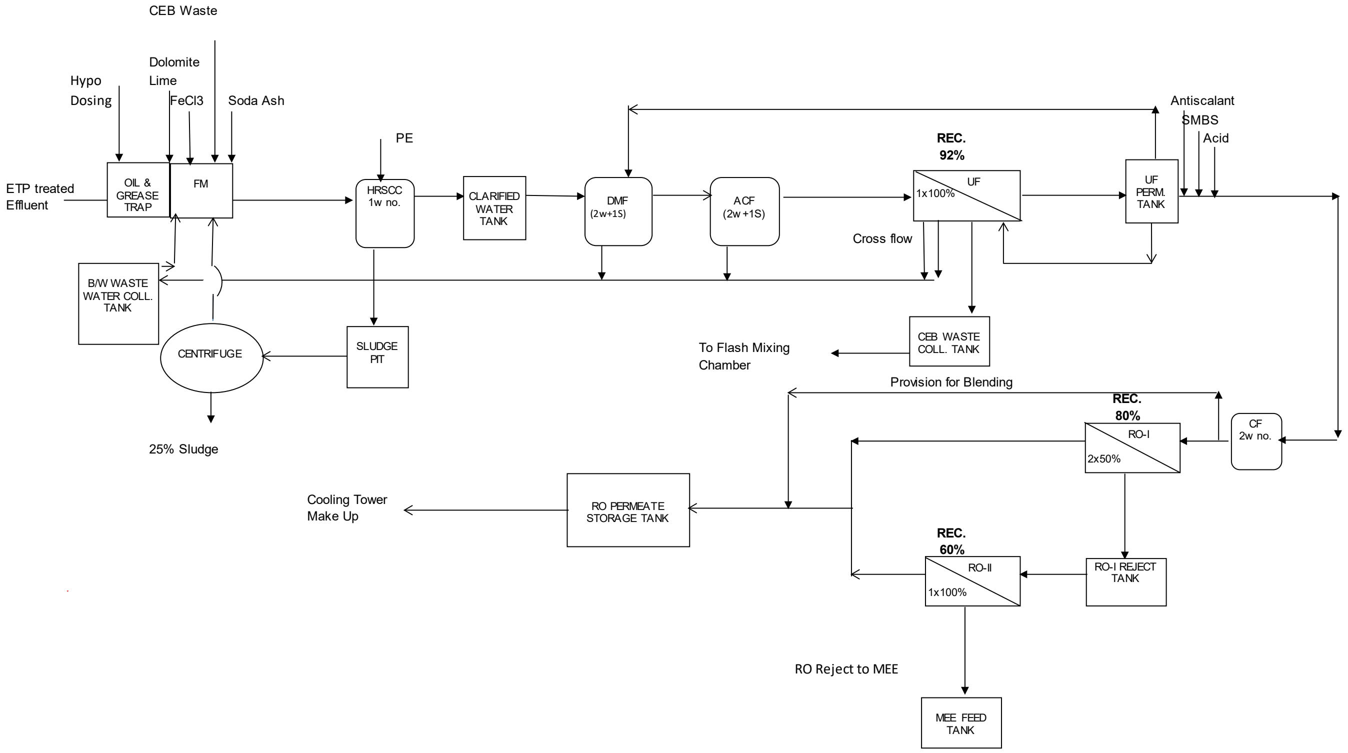

Fig. 7 Block Flow Diagram of RO Plant

Fig. 8 Overview of RO Plant

Table 11 Quality of Inlet and Treated Water of RO Plant

(*Clause* 2.2.2.14)

Sl No. (1)	Parameters (2)	Unit (3)	Inlet Quality (4)	RO-1 (5)	RO-2 (6)	RO-1 Reject (7)	Ro-2 Reject (8)
i)	RO permeate flow	m^3/hr	200	87	26.1	21.75	17.4
ii)	*p*H		8.4	6.8	7.0	8.4	8.7
iii)	Phosphate as PO4	mg/L	5.0	0.003	0.006	1.5	3.7
iv)	Zinc as Zn	mg/L	1.0				
v)	Total suspended Solids	mg/L	100				
vi)	Turbidity	NTU	60				
vii)	Total dissolved solids	mg/L	2600	68.94	138.85	14906	37111
viii)	Total hardness as CaCO3	mg/L	905			2113	5284
ix)	Total alkalinity as CaCO3	mg/L	126				
x)	Chloride as Cl	mg/L	637	25.251	50.784	3325	8247
xi)	Sulphates as SO4	mg/L	1250	11.7	23.863	6225	15547
xii)	Sodium as Na	mg/L	600	21.965	44.278	4066	10112
xiii)	Potassium as K	mg/L	50	1.653	3.322	244	606
xiv)	Iron as Fe	mg/L	0.4				
xv)	Oil and grease (free and floating)	mg/L	<10				
xvi)	Free chlorine	mg/L	Nil				
xvii)	Reactive silica as SiO2	mg/L	60	0.517	0.885	98	244
xviii)	COD	mg/L	100				

The concentrated sludge generated from RO and ZLD is sent to CPCB/ SPCB approved treatment facility (TSDF).

Block flow diagram of RO and ZLD plants are presented in Fig. 7 and 9 respectively. Photographs of RO and ZLD plants are shown in Fig. 8 and 10 respectively.

2.2.2.15 *Sanitary waste water*

Sanitary waste comes from the associated township and industrial premises. Sanitary waste from the plant and associated township is collected in pits at different locations within the plant and township area and pumped to the STP plant.

The treatment system requires removal of solids and other sanitary material from the effluent. For this purpose, a grit chamber is provided at the inlet of STP plant. Mostly for such industrial and associated township domestic sewage, an extended aeration with aerobic process is used. In this process, aerobic bacteria digest the pollutants. To establish an aerobic bacterial colony, it is necessary to provide air for the bacteria to breathe. In a sewage treatment plant, air is continuously supplied to the Biozone either by direct surface aeration using impellers propelled by pumps which whisk the surface of the liquid with air, or by submerged diffused aeration using blowers for air supply through bubble diffusers at the bottom of the tank. Aerobic conditions lead to an aerobic bacterial colony being established. These achieve almost complete oxidation and digestion of organic matter and organic pollutants to carbon dioxide, water and nitrogen, thus eliminating the odour and pollution problem. The effluent produced by this process is non-polluting and can be discharged to a watercourse or can be re-used in industrial process subject to quality compliance and with minor treatment such as chlorination.

After treatment in STP, treated effluent contains BOD less than 30 ppm, COD less than 250 ppm and TSS less than 100 mg/L.

The effluent generation and treatment scheme from an integrated ammonia-urea plant is depicted in Fig 11.

3 PHOSPHATIC FERTILIZERS

The phosphatic fertilizers can be categorized as straight phosphatic fertilizers having single nutrient P for example, SSP and TSP and Complex fertilizers having two or more nutrients. The basic raw materials used for production of phosphatic fertilizers are ammonia, sulphur, sulphuric acid, rock phosphate, phosphoric acid and nitric acid and muriate of potash (MOP). Single super phosphate is produced by reaction of rock phosphate with sulphuric acid. Triple super phosphate is produced as a reaction of rock phosphate with phosphoric acid. There is no production of TSP in the country. Complex fertilizer plants can follow two process routes namely, mixed acid route and nitro phosphate route. Mixed acid route is followed reaction of rock phosphate with phosphoric acid and in nitro-phosphate route rock phosphate reaction takes place with nitric acid.

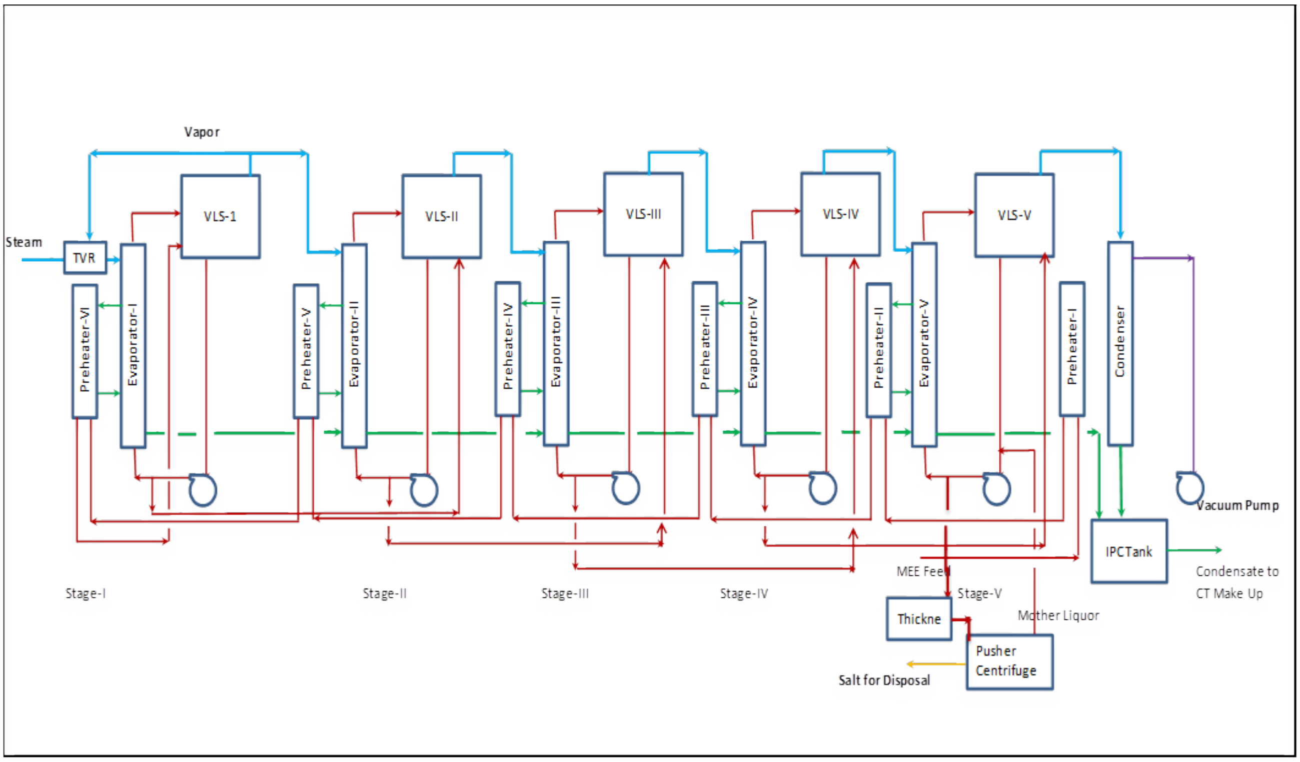

FIG. 9 BLOCK FLOW DIAGRAM OF ZLD PLANT

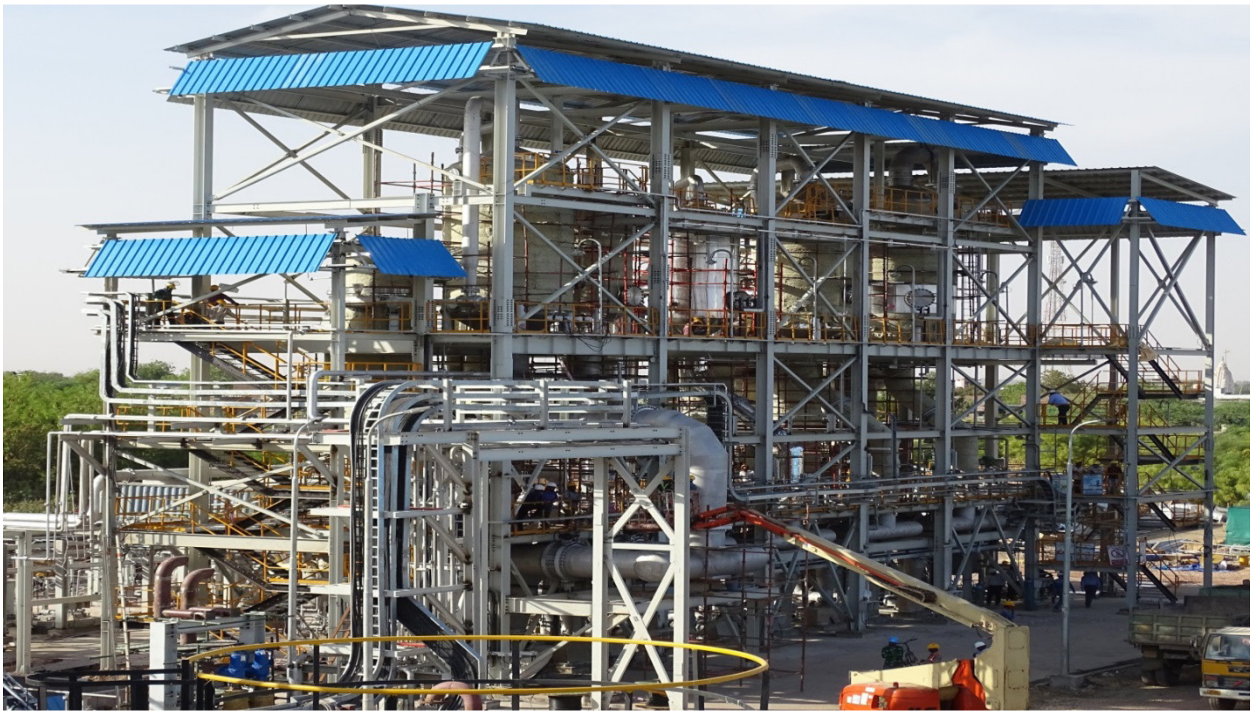

FIG. 10 OVERVIEW OF ZLD PLANT

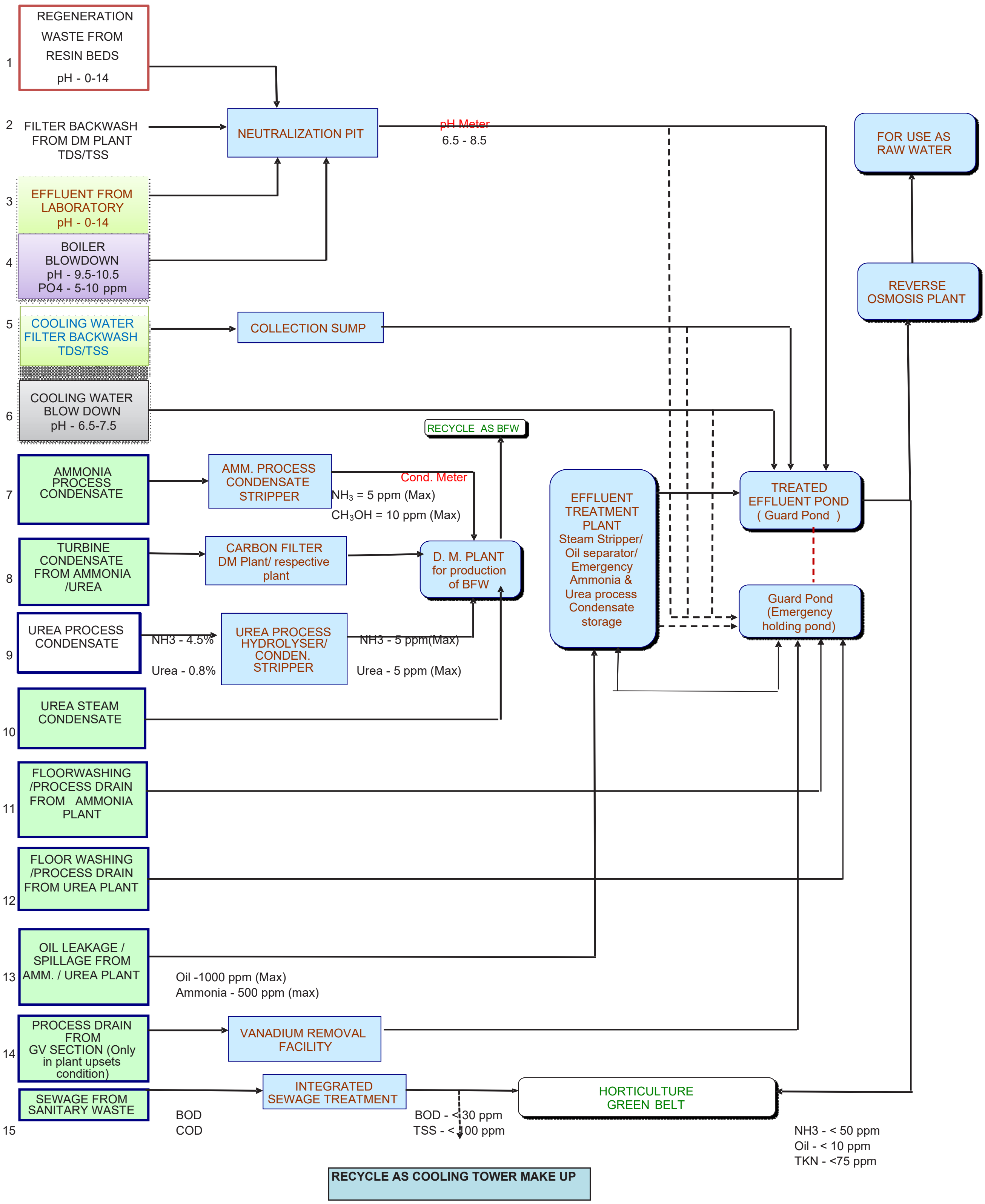
1
REGENERATION WASTE FROM RESIN BEDS
pH - 0-14
2
FILTER BACKWASH FROM DM PLANT TDS/TSS
NEUTRALIZATION PIT
pH Meter
6.5 - 8.5
FOR USE AS RAW WATER
3
EFFLUENT FROM LABORATORY
pH - 0-14
4
BOILER BLOWDOWN
pH - 9.5-10.5
PO4 - 5-10 ppm
REVERSE OSMOSIS PLANT
5
COOLING WATER FILTER BACKWASH TDS/TSS
COLLECTION SUMP
6
COOLING WATER BLOW DOWN
pH - 6.5-7.5
RECYCLE AS BFW
7
AMMONIA PROCESS CONDENSATE
AMM. PROCESS CONDENSATE STRIPPER
Cond. Meter
NH₃ = 5 ppm (Max)
CH₃OH = 10 ppm (Max)
EFFLUENT TREATMENT PLANT Steam Stripper/ Oil separator/ Emergency Ammonia & Urea process Condensate storage
TREATED EFFLUENT POND (Guard Pond)
8
TURBINE CONDENSATE FROM AMMONIA /UREA
CARBON FILTER DM Plant/ respective plant
D. M. PLANT for production of BFW
Guard Pond (Emergency holding pond)
9
UREA PROCESS CONDENSATE
NH3 - 4.5%
Urea - 0.8%
UREA PROCESS HYDROLYSER/ CONDEN. STRIPPER
NH3 - 5 ppm(Max)
Urea - 5 ppm (Max)
10
UREA STEAM CONDENSATE
11
FLOORWASHING /PROCESS DRAIN FROM AMMONIA PLANT
12
FLOOR WASHING /PROCESS DRAIN FROM UREA PLANT
13
OIL LEAKAGE / SPILLAGE FROM AMM. / UREA PLANT
Oil -1000 ppm (Max)
Ammonia - 500 ppm (max)
14
PROCESS DRAIN FROM GV SECTION (Only in plant upsets condition)
VANADIUM REMOVAL FACILITY
15
SEWAGE FROM SANITARY WASTE
BOD
COD
INTEGRATED SEWAGE TREATMENT
BOD - <30 ppm
TSS - <100 ppm
HORTICULTURE GREEN BELT
NH3 - < 50 ppm
Oil - < 10 ppm
TKN - <75 ppm
RECYCLE AS COOLING TOWER MAKE UP

FIG 11 TYPICAL EFFLUENT GENERATION AND TREATMENT SCHEME IN A NITROGENOUS FERTILIZER (AMMONIA-UREA) PLANT

3.1 Manufacturing

The plants can either produce acid at site or import.

3.1.1 *Manufacturing Process of Intermediate Acids*

There are three types of acid that are used as intermediate namely, sulphuric acid, phosphoric acid and nitric acid.

a) *Sulphuric Acid* — Sulphuric acid may be produced from elemental sulphur, pyrites (sulphide of iron), hydrogen sulphide as contained in sour gas from petroleum refineries, waste gas from burning of fuels and smelting operations, such as pyrites of copper, iron, zinc and lead. Elemental sulphur and, pyrites and sulphides are basic raw materials for sulphuric acid production.

Sulphur dioxide is first obtained by the burning of the molten sulphur in presence of air. Sulphur dioxide is then converted to sulphur trioxide in presence of vanadium pentoxide catalyst. The sulphur trioxide thus obtained is absorbed in recycling concentrated sulphuric acid in an absorption tower. The plants installed earlier and the smaller units of sulphuric acid plants use a single absorption process which has conversion efficiency of 96–98 percent. New large sulphuric acid production plants now–a–days utilize double conversion double absorption (DCDA) process. DCDA process can realize above 99 percent conversion efficiency.

$$S + O_2 \longrightarrow SO_2 \text{ (sulphur di-oxide)}$$

$$\underset{\longrightarrow}{2SO_2 + O_2\ V_2O_5 \text{ catalyst}}\ 2SO_3 \text{ (sulphur tri oxide)}$$

$$SO_3 + H_2O \longrightarrow H_2SO_4 \text{ (sulphuric acid)}$$

b) *Phosphoric Acid* — A high-grade rock phosphate and sulphuric acid are the raw materials for the production of phosphoric acid. There are two technologies which are used namely the wet process and the furnace process. Wet process is more viable commercially for a large plant.

Acidulation of finely ground rock phosphate with sulphuric acid to form phosphoric acid and gypsum. The gypsum is separated from the acid by filtration and acid is purified through removal of residual gypsum.

Concentration of phosphoric acid is done by evaporation to the desired concentration. Counter current washing of gypsum is employed to recover as much phosphoric acid as possible without acid dilution of acid. Dilute acid is recycled through the extraction step. By product gypsum is disposed of in dry or slurry form to gypsum ponds.

There are different processes of manufacturing of phosphoric acid namely, the di-hydrate process and the hemihydrate process. There is a difference of number of water molecule in gypsum. Quality of gypsum crystal determines the efficiency of filtration and loss of phosphate with gypsum. The other processes are combination of hemihydrate and dehydrate processes.

c) *Nitric Acid* — Essentially all the nitric acid produced is manufactured by oxidation of ammonia over a noble catalyst (platinum-rhodium). The ammonia is oxidized in presence of catalyst to form nitric oxide which further is oxidized into nitrogen di-oxide with excess air.

Liquid ammonia is evaporated, superheated and sent with compressed air to converter, containing platinum-rhodium catalyst gauges where ammonia oxidation takes place at a temperature of 850 deg. To 950 deg.C. In the converter ammonia is converted in to nitric oxide, which is then converted to nitrogen di-oxide in oxidation vessel with the help of secondary air. The process water absorbs nitrogen di-oxide to form nitric acid while running in counter flow to the rising nitrogen di-oxide in absorption column.

$$\underset{\text{ammonia}}{4\,NH_3} + \underset{\text{oxygen}}{5O_2} \longrightarrow \underset{\text{nitric oxide}}{4NO} + 6H_2O$$

$$2NO + O_2 \longrightarrow 2NO_2$$

$$3NO_2 + H_2O \longrightarrow NO + 2HNO_3 \text{ (nitric acid)}$$

3.1.2 *Single Super Phosphate*

Single superphosphate (SSP) is the most important single nutrient phosphatic fertilizer produced in India. It contains about 16 percent P_2O_5 in water soluble form. Raw materials required to produce SSP are rock phosphate and sulphuric acid. Imported as well as indigenous rock phosphate is used for SSP manufacture.

Rock phosphate (90 per cent passing through 100 mesh) of 30-35 per cent P_2O_5 content is mixed with sulphuric acid (65-70 per cent strength) in a specially designed mixer. The reaction is exothermic and proceeds in two steps. First, the sulphuric acid reacts with part of the rock forming phosphoric acid. Then the phosphoric acid formed reacts with more rock forming mono calcium phosphate. The two reactions take place concurrently but the first stage is completed rapidly while the second stage continues for several days or weeks. The fluid material from the mixer goes to a den where it solidifies. The solidification results from continued reaction and crystallization of mono calcium phosphate. In SSP production, phosphoric acid is found only as an intermediate. The super phosphate is excavated from the den after 30 min to 4 h. At this time, the superphosphate is still somewhat plastic and the temperature is around 100 deg. C. This product

$$\underset{\substack{\text{rock}\\\text{phosphate}}}{Ca_{10}(PO_4)\,F_2} + \underset{\substack{\text{sulphuric}\\\text{acid}}}{10\,H_2SO_4} + \underset{\text{water}}{20\,H_2O} \longrightarrow \underset{\substack{\text{phosphoric}\\\text{acid}}}{6H_3PO_4} + \underset{\text{gypsum}}{10\,(CaSO_4.2H_2O)} + \underset{\substack{\text{hydrogen}\\\text{fluoride}}}{2HF}$$

$$\underset{\substack{\text{rock}\\\text{phosphate}}}{Ca_{10}F_2(PO_4)_6} + \underset{\substack{\text{sulphuric}\\\text{acid}}}{7H_2SO_4} + \underset{\text{water}}{H_2O} \longrightarrow \underset{\substack{\text{mono}\\\text{calcium}\\\text{phosphate}}}{3Ca\,(H_2PO_4)_2.H_2O} + \underset{\substack{\text{calcium}\\\text{sulphate}}}{7CaSO_4} + \underset{\substack{\text{hydrogen}\\\text{fluoride}}}{2HF}$$

is removed from the den and conveyed to storage for final curing which requires 2-6 weeks depending on the nature and proportion of raw material and condition of manufacture. The cured product is reclaimed, milled, screened and either bagged for marketing or sent for granulation.

Granulation is done in the presence of steam in a drum or pan granulator and finished product is further send for packaging.

3.1.3 *Manufacturing of Complex Fertilizers (Di-Ammonium Phosphate)*

Compound or complex fertiliser means a fertiliser containing two or more nutrients during the production of which chemical reaction takes place. There are various grades of complex fertilizers depending on the nitrogen and phosphorus content in the product. DAP is the most common product and its production is discussed here. Other complex fertilizers are manufactured in similar way with different mole ratio of ammonia and phosphoric acid. Potassium is incorporated as third nutrient in form of MOP. As discussed earlier, complex fertilisers are produced by two different ways namely, mixed route and the nitro phosphate route.

a) *The Mixed Acid Route* — DAP is manufactured by reacting two moles of ammonia with one mole of phosphoric acid. Wet process phosphoric acid of about 40-42 per cent P_2O_5 is partially neutralized (to about 89 per cent) by anhydrous ammonia.

$$\underset{\text{ammonia}}{2NH_3} + \underset{\substack{\text{phosphoric}\\\text{Acid}}}{H_3PO_4} \longrightarrow \underset{\substack{\text{di-ammonium}\\\text{phosphate (DAP)}}}{(NH_4)_2HPO_4}$$

The preliminary neutralization is done in a pre-neutraliser and the mole ratio of NH_3:H_3PO_4 is maintained around 1.4. The resultant slurry containing a mixture of DAP and MAP is metered into the ammoniator granulator. Fines from recycle system are also added into the ammoniator granulator. Ammonia is further added into the bed of granules through special spargers to increase the mole ratio of NH_3:H_3PO_4 to around 2. Urea is also added to bring up ammonia to 18 per cent. The heat of reaction aids in removal of water vapour from the slurry. Over half of the water introduced in the process is evaporated in the pre-neutraliser and more water is driven off in the ammoniator granulator. Any unreacted ammonia gas is then scrubbed with water and weak phosphoric acid, and returned to the pre-neutraliser. The granulator discharge is then dried, screened after thorough drying, cooled and conditioned by coating agent, if necessary, and then bagged. Oversize granules are ground and recycled back to granulator along with fines.

Final product is further processed for packaging in the packaging plant.

b) *Nitro-phosphate Route* — In nitro-phosphate route, acidulation of rock takes place in presence of nitric acid in a series of reactors. The reaction mass contains calcium nitrate and phosphoric acid. The mixture can be converted into a solid granulated/prilled fertilizer by three or four important methods. Depending on the method adopted, the water soluble P_2O_5 content and the N-P ratio in the final product varies significantly.

$$Ca_{10}F_2(PO_4)_6 + 20\,HNO_3 \longrightarrow 6H_3PO_4 + 10\,Ca(NO_3)_2 + HF$$

In ODDA process route, to acidulated mixture requisite quantity of DAP or MAP is added to get desired 30 percent water soluble phosphate level. The reaction mass is ammoniated further to neutralization point resulting in formation of a slurry of ammonium nitrate, dicalcium phosphate and ammonium phosphate. Sufficient potassium is added to make the required grade. The reaction is further dried, granulated and screened.

In modified ODDA process, the acidulated mixture containing calcium nitrate and phosphoric acid is filtered to remove impurities and cooled for crystallization calcium nitrate. The cryastaline calcium nitrate which is hygroscopic is separated in centrifuge. After the separation, the remaining process liquor is ammoniated to yield high water soluble (up to 60 percent or more) phosphate containing nitrophosphate. The mix now contains ammonium nitrate, ammonium phosphate and dicalcium phosphate. The quantity of calcium nitrate separated decided the water soluble P_2O_5 in the product. To balance N:P_2O_5 ratio DAP/phosphoric acid is also added.

3.2 Effluent Generation and Treatment Scheme in Phosphatic Fertilizer Industries

3.2.1 *Water Consumption and Effluent Generation in SSP Plants*

By and large all SSP plants are having zero effluent discharge. This is provided as part of consent to operate condition. The waste water generated in the process during scrubbing of off gases from den is stored in tank made of concrete above ground level. The waste water containing fluoride is used for scrubbing of gases in scrubber.

3.2.2 *Water Consumption and Effluent Generation Quantity in Complex Fertilizer Plants*

The quantity of raw water consumed in the complex phosphatic fertilizer plant with phosphoric acid plant is in between 7.5 to 12.0 M^3/tonne of P_2O_5 while complex fertilizer plant without acid plant water consumption varies between 0 to 0.9 M^3/tonne of P_2O_5. The plant consuming zero raw water use treated water from the integrated ammonia-urea plant.

Quantity of treated effluent generated from complex fertilizer (with acid) plant varies between 0 to 0.90 M^3/tonne of P_2O_5.

The main source of effluents in phosphatic fertilizers are from scrubber liquors from various unit operations involving scrubbing of off-gases. Other sources of effluents are from Gypsum pond and floor washings in the process plants where all spills and leakages are mixed with water and run through process plant drains.

Waste water from complex fertilizer plants contains higher content of nutrients such as nitrates and phosphates. The entire wastewater generated from the complex fertilizer plant is reused in the scrubbers and process units. This helps in achieving zero process wastewater discharge operations.

There is no special treatment given for removal of nitrogen bearing components in the effluent from mixed acid based complex fertilizer plants.

Effluent from nitro phosphate based plants contains higher concentration of ammoniacal and nitrate nitrogen. The liquid effluent from nitro phosphate plant is first sent to the equalizing tanks to avoid shock loading and then subjected to air stripping to remove ammoniacal nitrogen. Finally, the nitrate is removed from the effluent from biological denitrification. A source of organic carbon is provided in the reactor and specially cultured bacteria breaks nitrate and nitrite to nitrogen and oxygen which escapes to atmosphere.

Main pollutants in phosphatic fertilizer plants are Phosphate and Fluorides. In effluent water fluorides are present as fluoro-silicic acid with small amounts of soluble salts as Sodium and Potassium fluorosilicates and hydrofluoric acid. Phosphorous is present as phosphoric acid with small amount of calcium phosphate.

For the removal of fluorides and phosphates, two stage treatment with Calcium carbonate and lime or double lime treatment is used.

3.2.3 *Process Description for ETP*

The combined effluent from process units of various sections passes through the effluent drains and is being collected locally in the central sump and pumped to effluent treatment plant (ETP). The effluent passes through an inlet chamber, bar screen, grit chamber and oil and grease trap prior to an equalization tank. The equalization tank is equipped with air diffusion system.

One dedicated facility for lime solution preparation is available with screen chamber, hoppers, belt conveyor, bucket elevator, screw conveyor and slurry preparation in concentrated lime preparation tanks, lime dosing tanks, lime dosing pumps and control valves. Here the lime powder with 80 ~ 86percent $Ca(OH)_2$ is being fed to the concentrated lime preparation tank and treated water added to make 20percent lime solution (approx.). This 20 percent lime solution is being taken to lime dosing tanks and it is diluted to 10 percent (approx.) by adding further quantity of treated water.

Effluent from equalization tank is pumped to flash mixing tank-1 where 10 percent lime solution is being added to raise the *p*H to 7 along with polyelectrolyte as a coagulant then passes to clari-flocculator – 1. Supernatant of clarifier-1 is passes to flash mixing tank-2 for second stage of lime treatment and then passes to clari-flocculator – 2. Where *p*H is raised to 10.5 by dosing further quantity of lime solution through control valve-2. The final clarified overflow from clarifier – 2 is being alkaline in nature with *p*H 10.5. *p*H of overflow water from clari-flocculator – 2 is corrected by adding sulphuric acid in pH correction tank where *p*H, phosphates, fluoride, TSS, TDS BOD, COD, oil and grease is achieved as per PCB norms. Treated water is used in ball mill of phosphoric acid plant.

The sludge from clari-flocculator – 1 and 2 is drained to Thickener and the thickened sludge is pumped to filter press for dewatering. Sludge cakes from the filter press are discharged to sludge storage yard which is used in the complex fertilizers plant as a filler. Treated water quality is being monitored by online *p*H meters.

3.2.4 *Theory of Chemical Treatment*

Phosphates is removed from wastewater by precipitation as calcium salt using calcium usually added in the form of lime that is, $Ca(OH)_2$. The reaction occurs primarily as follows:

$$3Ca^{2+} + 2(PO_4)^{3-} \longrightarrow Ca_3(PO_4)_2 \qquad (A)$$

When lime is added to water it reacts with the phosphates to precipitate calcium phosphate $Ca_3(PO_4)_2$. This reaction (A) is expected to dominate in the first stage of treatment till *p*H 7.0

However, the residual concentration of phosphates in water is higher than discharge limits due to partial solubility of calcium phosphate at neutral *p*H range. Therefore, second stage of phosphates removal at *p*H beyond 10 with lime is envisaged where calcium ions react with the residual phosphates to precipitate as hydroxylapatite $Ca_{10}(PO_4)_6(OH)_2$ governed by reaction (B).

$$10Ca^{2+} + 6PO_4^{3-} + 2OH^- \longrightarrow \underset{\text{(Hydroxylapatite)}}{Ca_{10}(PO_4)_6(OH)_2} \quad \text{(B)}$$

Chemistry of fluoride removal:

Calcium salts, including $Ca(OH)_2$, $CaSO_4$, and $CaCl_2$ are used to precipitate fluorides as insoluble CaF_2. The precipitation of fluoride using lime occurs as follows:

$$Ca(OH)_2 + 2HF \longrightarrow CaF_2 \downarrow + 2H_2O$$

Stoichiometrically, the precipitation should affect the reduction of fluoride levels to the limit of solubility of fluoride in the CaF_2 system. But the equilibrium that affects the solubility of fluoride includes not only the CaF_2 itself but also the species $H^{2+}F$ and HF^{2-} as well as the ion-pair CaF^+. This results in presence of traces of fluoride after initial precipitation meriting a second stage of fluoride removal. The second stage is affected by chemisorption of fluoride on calcium phosphate and by adsorption on hydroxylapatite.

The presence of alkalinity and precipitable phosphates enhance the precipitation of fluoride in which the carbonate radical of the apatite complex, $Ca(PO4)_6.CaCO_3$, is replaced by fluoride to form an insoluble fluorapatite:

$$Ca(PO_4)_6.CaCO_3 + 2F^- \longrightarrow Ca(PO_4)_6.CaF_2 \downarrow + CO_2^{3-} \quad \text{(D)}$$

$$Ca(PO_4)_6 + 2CaF \longrightarrow \underset{\text{(Fluorapatite)}}{Ca(PO_4)_6.CaF_2 \downarrow} \quad \text{(E)}$$

Since, the precipitation of phosphate is being carried out simultaneously the process of co precipitation takes over in the second stage of treatment resulting in highly efficient removal of fluorides from wastewater. Table 12 provides the quality of effluent at the outlet of ETP.

Table 12 Typical Quality of Final Outlet of ETP

(*Clause* 3.2.4)

Sl No. (1)	Parameters (2)	ETP Outlet (3)
i)	*p*H	6.5 – 8.5
ii)	Fluoride as F	10 ppm *Max.*
iii)	Phosphate as P	20 ppm *Max.*
ix)	Oil and grease	10 mg/lit *Max.*
v)	Total suspended solids	100 mg/lit *Max*
vi)	Nitrate nitrogen as N	20 ppm *Max.*
vii)	Total ammonical nitrogen as N	50 ppm *Max.*
viii)	Total Kjheldahl nitrogen as N	75 ppm *Max.*
ix)	Free ammonical nitrogen	4.0ppm *Max.*

4 SAMPLING AND ANALYTICAL CONTROL

In order to ensure that all effluent treatment facilities are performing to achieve the desired results, a sampling and testing plan is very important. An environmental monitoring laboratory is provided in the industry which sets up the sampling plan with optimum frequency and establishes standard sampling and testing procedures. Suitable instrumentation for testing of untreated and treated effluents are made available.

Suitable automatic online continuous monitoring system of pollutants is installed to monitor parameters such as *p*H and ammonia and these are connected to the SPCB/CPCB through available software for proper monitoring by statutory authorities.

5 GUIDELINES FOR UTILISATION OF TREATED EFFLUENT IN IRRIGATION

The Central Pollution Control Board (CPCB) has issued a guidelines for utilization of treated effluent in irrigation. The guidelines define the zero liquid discharge (ZLD) as “zero liquid discharge (ZLD) implies that the industries are not discharging any effluent, either on the land or in the water body or at any other place that is, recycling the same in the process entirely without releasing any effluent. ZLD accomplishment may need physical and chemical treatment, followed by biological system to remove organic load. The treated effluents can be then subjected for concentration and evaporation. The concentration method quite often involves the adoption of reverse osmosis (RO) and nano filtration (NF) methods. The evaporation methods involve drying/evaporation of effluent in multi effect evaporators (MEE).

It was recognized that adopting ZLD practices may not be feasible in many cases in view of techno-economic reasons. However, the industries should still to be encouraged for recycling and reuse of waste water as far as practicable in order to minimize the fresh water consumption and discharge of waste water into the environment. The treated waste water of an industry may also be utilized for irrigation. This type of utilisation/application is considered an efficient approach for managing/conserving water resources, compensating water shortages caused by seasonality or the irregular availability of water sources for irrigation throughout the year.

The possible risks of wastewater usage in agriculture may range from changes to physico-chemical and micro-biological properties of soils to impact on human health. In unfavorable economic conditions, the search for alternative irrigation sources, such as the use of untreated or inadequately treated wastewater may result in risk factors. Thus, it is necessary to ensure the beneficial aspects of this practice before application of treated wastewater in irrigation.

The guidelines for utilisation of effluent in irrigation requires engagement of an agricultural scientist or tie-up with an agricultural university or institute for advice on the utilization or the rate of application of the effluent for irrigation considering the agro-climatic conditions. Industry is required to prepare a comprehensive irrigation management plan (IMP), in consultation with the agricultural scientist or agriculture university/institute and submit to SPCBs/PCCs which should verify the same while issuing Consent to the industry.

The treated effluent should meet the norms prescribed for irrigation under Environment (Protection) Rules, 1986/Consent. The effluent should also conform to total dissolved solid (TDS)- 2100 mg/L and sodium adsorption ratio (SAR)- preferably less than 18 but not more than 26, depending on soil/crop type, besides meeting any other parameters suggested by agricultural scientist or agricultural university/institute in the IMP.

The utilization of treated waste water should also consider conditions such as crop type, climate, irrigation type, soil condition, soil permeability and total salt concentration. The command area for effluent utilisation should be as near as feasible to the industry in order to facilitate easy monitoring and effective control.

There are other requirements with respect to capacity of storage tank of treated effluent, distribution network of impervious conduits to cover the irrigated area, analysis of treated effluent periodically (15 days), analysis of soil under irrigation twice a year and monitoring of ground water quality twice a year from NABL/EPA/SPCBs/PCCs recognized/accredited laboratories. Reports regarding compliance of effluent quality standards and status of soil and ground water quality shall be submitted to SPCBs/PCCs twice in a year, in first week of January and July.

In case of observation of any deterioration of the soil and groundwater quality parameters in the assessment by agricultural scientist or agricultural university/institute, the application of effluent should be stopped immediately and the industry should inform the SPCB, accordingly. The industry shall be solely responsible for reclaiming the soil and water quality at their cost in the affected area.

ANNEX A

(*Forward*)

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ANNEX B

(*Foreword*)

COMMITTEE COMPOSITION

Environment Protection Sectional Committee, CHD 32

Organization	*Representative(s)*
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